

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION
(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 30 April 2001 (30.04.01)	Applicant's or agent's file reference P1999S004
International application No. PCT/EP00/07910	Priority date (day/month/year) 17 August 1999 (17.08.99)
International filing date (day/month/year) 11 August 2000 (11.08.00)	
Applicant HOLT, David, Gary, Lawton	

1. The designated Office is hereby notified of its election made:

in the demand filed with the International Preliminary Examining Authority on:

13 March 2001 (13.03.01)

in a notice effecting later election filed with the International Bureau on:

2. The election was

was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer Jean-Marie McAdams
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
22 February 2001 (22.02.2001)

PCT

(10) International Publication Number
WO 01/12762 A1

(51) International Patent Classification⁷: **C10M 163/00 //**
(C10M 163/00, 159;12, 129;42, 129;93)

Lawton [GB/US]; 18 Saddler Drive, Medford, NJ 08055
(US).

(21) International Application Number: **PCT/EP00/07910**

(74) Agents: DEW, Melvyn, John et al.; ExxonMobil Chemical Europe Inc., P.O. Box 105, B-1830 Machelen (BE).

(22) International Filing Date: 11 August 2000 (11.08.2000)

(81) Designated States (national): CA, JP, SG, US.

(25) Filing Language: English

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(26) Publication Language: English

Published:

— With international search report.

(71) Applicant (for all designated States except US): **EXXON-MOBIL RESEARCH AND ENGINEERING COMPANY [US/US]**; 1545 Route 22 East, Clinton Township, Annandale, NJ 08801 (US).

— Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.

(72) Inventor; and
(75) Inventor/Applicant (for US only): HOLT, David, Gary,

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 01/12762 A1

(54) Title: CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS

(57) Abstract: Lubricating oil formulations comprising base oil, such sulfur-phosphorous anti-wear/extreme pressure agents and such hindered phenol antioxidants which anti-wear/extreme-pressure agents and hindered phenolic antioxidants are prone to react and form crystals wherein the base oil is characterized as having a saturates content of less than 99 % which base oil is stabilized against the above mentioned crystal formation by the addition of a minor amount of a high molecular weight di- or polycarboxylic acid anhydride, or mixture thereof.

IPSS updated
18 SEP 2000
K. STORMS

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference
(if desired, (12 characters maximum))

P1999S004

Box No. I TITLE OF INVENTION

STABILIZED LUBRICATING FORMULATION AND METHOD

Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity: full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

EXXON RESEARCH AND ENGINEERING COMPANY
Exxon Mobil Corporation
Downstream Law, Research and Engineering
1545 Route 22 East
Annandale, NJ 08801-0900
USA

This person is also inventor.

Telephone No.

Faximile No.

Teleprinter No.

State (that is, country) of nationality: US

State (that is, country) of residence: US

This person is applicant all designated States all designated States except the United States of America the United States of America only the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity: full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

HOLT, David Gary Lawton
18 Saddler Drive
Medford, NJ 08055
USA

This person is:

applicant only

applicant and inventor

inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality: UK

State (that is, country) of residence: US

This person is applicant all designated States all designated States except the United States of America the United States of America only the States indicated in the Supplemental Box

Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE: OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:

agent

common representative

Name and address: (Family name followed by given name; for a legal entity: full official designation. The address must include postal code and name of country.)

DEW, Melvyn John ; MARESCHAL, Anne M. ; VELDHUIZEN, Albert Dirk Willem
ExxonMobil Chemical Europe Inc.
P.O. BOX 105
B-1830 MACHELEN
BELGIUM

Telephone No.

+32-2-722.2268

Faximile No.

+32-2-722.2299

Teleprinter No.

Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

Regional Patent

- AP ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, MZ Mozambique, SD Sudan, SL Sierra Leone, SZ Swaziland, TZ United Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- EA Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- EP European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- OA OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)

National Patent (if other kind of protection or treatment desired, specify on dotted line):

- | | | |
|---|--|--|
| <input type="checkbox"/> AE United Arab Emirates | <input type="checkbox"/> LC Saint Lucia | |
| <input type="checkbox"/> AG Antigua and Barbuda | <input type="checkbox"/> LK Sri Lanka | |
| <input type="checkbox"/> AL Albania | <input type="checkbox"/> LR Liberia | |
| <input type="checkbox"/> AM Armenia | <input type="checkbox"/> LS Lesotho | |
| <input type="checkbox"/> AT Austria | <input type="checkbox"/> LT Lithuania | |
| <input type="checkbox"/> AU Australia | <input type="checkbox"/> LU Luxembourg | |
| <input type="checkbox"/> AZ Azerbaijan | <input type="checkbox"/> LV Latvia | |
| <input type="checkbox"/> BA Bosnia and Herzegovina | <input type="checkbox"/> MA Morocco | |
| <input type="checkbox"/> BB Barbados | <input type="checkbox"/> MD Republic of Moldova | |
| <input type="checkbox"/> BG Bulgaria | <input type="checkbox"/> MG Madagascar | |
| <input type="checkbox"/> BR Brazil | <input type="checkbox"/> MK The former Yugoslav Republic of Macedonia | |
| <input type="checkbox"/> BY Belarus | <input type="checkbox"/> MN Mongolia | |
| <input type="checkbox"/> BZ Belize | <input type="checkbox"/> MW Malawi | |
| <input checked="" type="checkbox"/> CA Canada | <input type="checkbox"/> MX Mexico | |
| <input type="checkbox"/> CH and LI Switzerland and Liechtenstein | <input type="checkbox"/> MZ Mozambique | |
| <input type="checkbox"/> CN China | <input type="checkbox"/> NO Norway | |
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| <input type="checkbox"/> CZ Czech Republic | <input type="checkbox"/> PT Portugal | |
| <input type="checkbox"/> DE Germany | <input type="checkbox"/> RO Romania | |
| <input type="checkbox"/> DK Denmark | <input type="checkbox"/> RU Russian Federation | |
| <input type="checkbox"/> DM Dominica | <input type="checkbox"/> SD Sudan | |
| <input type="checkbox"/> DZ Algeria | <input type="checkbox"/> SE Sweden | |
| <input type="checkbox"/> EE Estonia | <input checked="" type="checkbox"/> SG Singapore | |
| <input type="checkbox"/> ES Spain | <input type="checkbox"/> SI Slovenia | |
| <input type="checkbox"/> FI Finland | <input type="checkbox"/> SK Slovakia | |
| <input type="checkbox"/> GB United Kingdom | <input type="checkbox"/> SL Sierra Leone | |
| <input type="checkbox"/> GD Grenada | <input type="checkbox"/> TJ Tajikistan | |
| <input type="checkbox"/> GE Georgia | <input type="checkbox"/> TM Turkmenistan | |
| <input type="checkbox"/> GH Ghana | <input type="checkbox"/> TR Turkey | |
| <input type="checkbox"/> GM Gambia | <input type="checkbox"/> TT Trinidad and Tobago | |
| <input type="checkbox"/> HR Croatia | <input type="checkbox"/> TZ United Republic of Tanzania | |
| <input type="checkbox"/> HU Hungary | <input type="checkbox"/> UA Ukraine | |
| <input type="checkbox"/> ID Indonesia | <input type="checkbox"/> UG Uganda | |
| <input type="checkbox"/> IL Israel | <input checked="" type="checkbox"/> US United States of America | |
| <input type="checkbox"/> IN India | <input type="checkbox"/> UZ Uzbekistan | |
| <input type="checkbox"/> IS Iceland | <input type="checkbox"/> VN Viet Nam | |
| <input checked="" type="checkbox"/> JP Japan | <input type="checkbox"/> YU Yugoslavia | |
| <input type="checkbox"/> KE Kenya | <input type="checkbox"/> ZA South Africa | |
| <input type="checkbox"/> KG Kyrgyzstan | <input type="checkbox"/> ZW Zimbabwe | |
| <input type="checkbox"/> KP Democratic People's Republic of Korea | Check-box reserved for designating States which have become party to the PCT after issuance of this sheet: | |
| <input type="checkbox"/> KR Republic of Korea | <input type="checkbox"/> | |
| <input type="checkbox"/> KZ Kazakhstan | | |

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)

Box No. VI PRIORITY CLAIM		<input type="checkbox"/> Further priority claims indicated in the Supplemental Box.		
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application: regional Office	international application: receiving Office
item (1) 17 AUGUST 1999	9919490.4	UK		
item (2)				
item (3)				

The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s):

* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used): ISA / EP	Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority): Date (day/month/year) Number Country (or regional Office)		
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Box No. VIII CHECK LIST; LANGUAGE OF FILING	
This international application contains the following number of sheets: request : 3 description (excluding sequence listing part) : 15 claims : 3 abstract : 1 drawings : sequence listing part of description : Total number of sheets : 22	This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input type="checkbox"/> separate signed power of attorney 3. <input checked="" type="checkbox"/> copy of general power of attorney; reference number, if any: 55, 18826 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input type="checkbox"/> other (specify): Acknowledgement of receipt form

Figure of the drawings which should accompany the abstract:	Language of filing of the international application: EN
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Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Anne Mareschal

General Authorization No. 55

For receiving Office use only	
1. Date of actual receipt of the purported international application:	2. Drawings: <input type="checkbox"/> received: <input type="checkbox"/> not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:	
4. Date of timely receipt of the required corrections under PCT Article 11(2):	
5. International Searching Authority (if two or more are competent): ISA /	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.

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Date of receipt of the record copy by the International Bureau:	

This sheet is not part of the international application.

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FEE CALCULATION SHEET Annex to the Request

For receiving Office use only

International application No.

Applicant's or agent's file reference P1999S004

Date stamp of the receiving Office

Applicant

EXXON RESEARCH AND ENGINEERING COMPANY

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE EUR 102

T

2. SEARCH FEE EUR 945

S

International search to be carried out by EP

(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FEE

Basic Fee

The international application contains 22 sheets.

first 30 sheets EUR 413

b1

remaining sheets x additional amount = b2

Add amounts entered at b1 and b2 and enter total at B 413

B

Designation Fees

The international application contains 5 designations.

5 x 95 = 475

D

number of designation fees payable (maximum 8) amount of designation fee

Add amounts entered at B and D and enter total at I EUR 888

I

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicant is (or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT (if applicable)

P

5. TOTAL FEES PAYABLE EUR 1935

TOTAL

The designation fees are not paid at this time.

MODE OF PAYMENT

authorization to charge deposit account (see below)
 cheque
 postal money order

bank draft
 cash
 revenue stamps

coupons
 other (specify):

DEPOSIT ACCOUNT AUTHORIZATION (this mode of payment may not be available at all receiving Offices)

The RO EP is hereby authorized to charge the total fees indicated above to my deposit account.

(this check-box may be marked only if the conditions for deposit accounts of the receiving Office so permit) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

28300171

11 August 2000

Signature

[Signature]

55 (rev).

1 ALLGEMEINE VOLLMACHT
GENERAL AUTHORISATION
POUVOIR GENERAL

2 Ich (Wir) / I (We) / Je (Nous)

ExxonMobil Research and Engineering Company,
Florham Park,
New Jersey,
United States of America,
Incorporated in the State of Delaware

3 bevollmächtige(n) hiermit / do hereby authorise / autorise (autorisons) par la présente

SOMERS, Harold Arnold

FLETCHER WATTS, Susan Jane

of: Esso Engineering (Europe) Ltd., Patents and Licences, Mailpoint 70, Esso House, Ermyn Way, Leatherhead, Surrey KT22 8XE. United Kingdom.

DEW, Melvyn John

of: Exxon Chemical Europe Inc., Hermeslaan 2, B-1831, Machelen, Belgium.

4 mich (uns) in den durch das Europäische Patentübereinkommen geschaffenen Verfahren in allen meinen (unseren) Patentangelegenheiten zu vertreten, alle Handlungen für mich (uns) vorzunehmen und Zahlungen für mich (uns) in Empfang zu nehmen.
to represent me (us) in all proceedings established by the European Patent Convention and to act for me (us) in all patent transactions and to receive payments on my (our) behalf.
à me (nous) représenter pour ce qui concerne toutes mes (nos) affaires de brevet dans toute procédure instituée par la Convention sur le brevet européen et, à ce titre, à agir en mon (notre) nom et à recevoir des paiements pour mon (notre) compte.

Die Vollmacht gilt auch für Verfahren nach dem Vertrag über die Internationale Zusammenarbeit auf dem Gebiet des Patentwesens.
This authorisation shall also apply to the same extent to any proceedings established by the Patent Cooperation Treaty.
Ce pouvoir s'applique également à toute procédure instituée par le Traité de coopération en matière de brevets.

Weitere Vertreter sind auf einem gesonderten Blatt angegeben. / Additional representatives indicated on supplementary sheet.
Les autres mandataires sont mentionnés sur une feuille supplémentaire.

5 Untervollmacht kann erteilt werden. / Sub-authorisation may be given. / Le pouvoir pourra être délégué.

6 Bitte die gelbe Kopie, ergänzt um die Nr. der allgemeinen Vollmacht, an den Vollmachtgeber zurücksenden.
Please return the yellow copy, supplemented by the General Authorisation No., to the authorisor.
Prié de renvoyer la copie jaune au mandant, munie du n° du pouvoir général.

Ort/Place/Lieu Leatherhead, Surrey, GB. Datum/Date 07 January 2000
Unterschrift(en) / Signature(s)

HAROLD ARNOULD SOMERS / *Omnes* - Authorised signatory

7 Das Formblatt muß vom (von den) Vollmachtgeber(n) (bei juristischen Personen vom Unterschriftberechtigten) eigenhändig unterzeichnet sein. Nach der Unterschrift bitte den (die) Namen des (der) Unterzeichneten mit Schreibmaschine wiederholen (bei Juristischen Personen die Stellung des Unterschriftsberechtigten innerhalb der Gesellschaft angeben).
The form must bear the personal signature(s) of the authorisor(s) (in the case of legal persons, that of the officer empowered to sign). After the signature, please type the name(s) of the signatory(ies) adding, in the case of legal persons, his (their) position within the company.

Le formulaire doit être signé de la propre main du (des) mandant(s) (dans le cas de personnes morales, de la personne ayant qualité pour signer). Veuillez ajouter à la machine, après la signature, le (les) nom(s) du (des) signataire(s) en mentionnant, dans le cas de personnes morales, ses (leurs) fonctions au sein de la société.

Bitte übermitteln die 3 Blätter 1004.1-3 dem EPA in München, Direktion 5.1.1, und beachten Sie Blatt 1004.4, insbesondere Nr.3 b.
Please forward the 3 sheets 1004.1-3 to the EPO at Munich, Directorate 5.1.1 and pay attention to sheet 1004.4, especially point 3 b.
Prière de faire parvenir les 3 feuilles 1004.1-3 à l'OEB à Munich, Direction 5.1.1 et de tenir compte de la feuille 1004.5, notamment au point 3 b.

ALLGEMEINE VOLLMACHT
GENERAL AUTHORISATION
POUVOIR GENERAL

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Cadre réservé à l'administration Sub
Nr. der allgemeinen Vollmacht / General Authorisation No.
Nº du pouvoir général

55

2 Ich (Wir) / I (We) / Je (Nous)

EXXONMOBIL RESEARCH & ENGINEERING COMPANY
180 Park Avenue
Florham Park, NJ 07932
U.S.A.

3 bevollmächtige(n) hiermit / do hereby authorise / autorise (autorisons) par la présente

Anne MARESCHAL,
having a professional address at
Exxon Chemical Europe Inc., Law Technology, Hermeslaan 2, B-1831 Machelen, Belgium

4 mich (uns) in den durch das Europäische Patentübereinkommen geschaffenen Verfahren in allen meinen (unseren) Patentangelegenheiten zu vertreten, alle Handlungen für mich (Uns) vorzunehmen und Zahlungen für mich (Uns) in Empfang zu nehmen.

to represent me (us) in all proceedings established by the European Patent Convention and to act for me (us) in all patent transactions and to receive payments on my (our) behalf.

à me (nous) représenter pour ce qui concerne toutes mes (nos) affaires de brevet dans toute procédure instituée par la Convention sur le brevet européen et, à ce titre, à agir en mon (notre) nom et à recevoir des paiements pour mon (notre) compte.

Die Vollmacht gilt auch für Verfahren nach dem Vertrag über die internationale Zusammenarbeit auf dem Gebiet des Patentwesens.
This authorisation shall also apply to the same extent to any proceedings established by the Patent Cooperation Treaty.
Ce pouvoir s'applique également à toute procédure instituée par le Traité de coopération en matière de brevets.

Weitere Vertreter sind auf einem gesonderten Blatt angegeben. / Additional representatives indicated on supplementary sheet.
Les autres mandataires sont mentionnés sur une feuille supplémentaire.

5 Untervollmacht kann erteilt werden. / Sub-authorisation may be given. / Le pouvoir pourra être délégué.

6 Bitte die gelbe Kopie, ergänzt um die Nr. der allgemeinen Vollmacht, an den Vollmachtgeber zurücksenden.
Please return the yellow copy, supplemented by the General Authorisation No., to the authorisor.
Prière de renvoyer la copie jaune au mandant, munie du n° du pouvoir général.

Ort/Place/Lieu

Machelen, Belgium

Datum / Date

22 May 2000

Unterschrift(en) / Signature(s)

Melvyn John DEW
Authorised Signatory

7 Das Formblatt muß vom (von den) Vollmachtgeber(n) (bei juristischen Personen vom Unterschriftsberechtigten) eigennäig unterzeichnet sein. Nach der Unterschrift bitte den (die) Namen des (der) Unterzeichneten mit Schreibmaschine wiederholen (bei juristischen Personen die Stellung des Unterschriftsberechtigten innerhalb der Gesellschaft angeben).

The form must bear the personal signature(s) of the authorisor(s). (In the case of legal persons, that of the officer empowered to sign). After the signature, please type the name(s) of the signatory(ies) adding, in the case of legal persons, his (their) position within the company.

Le formulaire doit être signé de la propre main du (des) mandant(s) (dans le cas de personnes morales, de la personne ayant qualité pour signer). Veuillez ajouter à la machine, après la signature, le (les) nom(s) du (des) signataire(s) en mentionnant, dans le cas de personnes morales, ses (leurs) fonctions au sein de la société.

GENERAL AUTHORISATION¹

For official use only
General Authorisation N°

18826

I (We)²

EXXON RESEARCH AND ENGINEERING COMPANY, a Corporation duly organized and existing under the laws of the State of Delaware, United States of America

PO Box 390, Florham Park, New Jersey 07932,
United States of America

do hereby authorise³

VELDHUIZEN, Albert Dirk Willem

Exxon Chemical Limited
Exxon Chemical Technology Centre
PO Box 1
Abingdon
Oxfordshire OX13 6BB
United Kingdom

(Additional representatives indicated on supplementary sheet).

to represent me (us) in all proceedings established by the European Patent Convention and to act for me (us) in all patent transactions and to receive payments on my (our) behalf.

Substitute authorisation may be given.

Place Abingdon, United Kingdom

Date 26 November 1987

Signature(s)⁴



BAWDEN, Peter Charles - Authorised Signatory
Chief European Patent Attorney
- Please supplement signature(s) by typewritten name(s) -

PATENT COOPERATION TREATY

From the RECEIVING OFFICE

PCT

To:

Dew, Melvyn John
 EXXONMOBIL CHEMICAL EUROPE INC.
 P.O. Box 105
 B-1830 Machelen
 BELGIQUE

**NOTIFICATION OF THE INTERNATIONAL
APPLICATION NUMBER AND OF THE
INTERNATIONAL FILING DATE**

(PCT Rule 20.5(c))

Date of mailing
(day/month/year)

04 OCT 2000

Applicant's or agent's file reference
P1999S004

IMPORTANT NOTIFICATION

International application No.
PCT/ EP 00/ 07910

International filing date (day/month/year)
11/08/2000

Priority date (day/month/year)
17/08/1999

Applicant
EXXON RESEARCH AND ENGINEERING COMPANY

Title of the invention

1. The applicant is hereby notified that the international application has been accorded the international application number and the international filing date indicated above.
2. The applicant is further notified that the record copy of the international application was transmitted to the International Bureau on the above date of mailing.
3. Other:

* The International Bureau monitors the transmittal of the record copy by the receiving Office and will notify the applicant (with Form PCT/IB/301) of its receipt. Should the record copy not have been received by the expiration of 14 months from the priority date, the International Bureau will notify the applicant (Rule 22.1(c)).

Name and mailing address of the receiving Office

 European Patent Office, P.B. 5818 Patentlaan 2
 NL-2280 HV Rijswijk
 Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
 Fax: (+ 31-70) 340-3016

Authorized officer



G.A.B. VAN FRAASSEN

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF RECEIPT OF
RECORD COPY

(PCT Rule 24.2(a))

From the INTERNATIONAL BUREAU

To:

DEW, Melvyn, John
 ExxonMobil Chemical Europe Inc.
 P.O. Box 105
 B-1830 Maelbeek
 BELGIQUE

Date of mailing (day/month/year) 31 October 2000 (31.10.00)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference P1999S004	International application No. PCT/EP00/07910

The applicant is hereby notified that the International Bureau has received the record copy of the international application as detailed below.

Name(s) of the applicant(s) and State(s) for which they are applicants:

EXXON RESEARCH AND ENGINEERING COMPANY (for all designated States except US)
 HOLT, David, Lawton (for US)

International filing date : 11 August 2000 (11.08.00)
 Priority date(s) claimed : 17 August 1999 (17.08.99)
 Date of receipt of the record copy by the International Bureau : 09 October 2000 (09.10.00)
 List of designated Offices :
 EP :AT,BE,CH,CY,DE,DK,ES,FI,FR,GB,GR,IE,IT,LU,MC,NL,PT,SE
 National :CA,JP,SG,US

ATTENTION

The applicant should carefully check the data appearing in this Notification. In case of any discrepancy between these data and the indications in the international application, the applicant should immediately inform the International Bureau.

In addition, the applicant's attention is drawn to the information contained in the Annex, relating to:

- time limits for entry into the national phase
- confirmation of precautionary designations
- requirements regarding priority documents

A copy of this Notification is being sent to the receiving Office and to the International Searching Authority.

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 34, chemin des Colombettes
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Authorized officer:

Athina Nickitas-Etienne

Telephone No. (41-22) 338.83.38

003621111

IPSS UPDATED PATENT COOPERATION TREATY
K. STORMS

19 JAN 2001 PCT

**NOTIFICATION OF THE RECORDING
OF A CHANGE**

(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)

Date of mailing (day/month/year)
09 January 2001 (09.01.01)

From the INTERNATIONAL BUREAU

To:

DEW, Melvyn, John
ExxonMobil Chemical Europe Inc.
P.O. Box 105
B-1830 Machelen
BELGIQUE

Applicant's or agent's file reference	IMPORTANT NOTIFICATION
P1999S004	
International application No.	International filing date (day/month/year)
PCT/EP00/07910	11 August 2000 (11.08.00)

1. The following indications appeared on record concerning:

the applicant the inventor the agent the common representative

Name and Address	State of Nationality	State of Residence
EXXON RESEARCH AND ENGINEERING COMPANY Exxon Mobil Corporation Downstream Law, Research and Engineering 1545 Route 22 East Annandale, NJ 08801-0900 United States of America	US	US
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

the person the name the address the nationality the residence

Name and Address	State of Nationality	State of Residence
EXXONMOBIL RESEARCH AND ENGINEERING COMPANY 1545 Route 22 East Clinton Township Annandale, NJ 08801 United States of America	US	US
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:

4. A copy of this notification has been sent to:
<input checked="" type="checkbox"/> the receiving Office <input type="checkbox"/> the designated Offices concerned
<input checked="" type="checkbox"/> the International Searching Authority <input type="checkbox"/> the elected Offices concerned
<input type="checkbox"/> the International Preliminary Examining Authority <input type="checkbox"/> other:

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer Athina Nickitas-Etienne Telephone No.: (41-22) 338.83.38
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19.01.01
19.01.01

PATENT COOPERATION TREATY

1) To KS/NC FOR DE

Kaohsiung

22 MAR. 2001

PCT

NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

From the INTERNATIONAL BUREAU

To:

DEW, Melvyn, John
 ExxonMobil Chemical Europe Inc.
 P.O. Box 105
 B-1830 Machelen
 BELGIQUE

Date of mailing (day/month/year)

22 February 2001 (22.02.01)

Applicant's or agent's file reference

P1999S004

IMPORTANT NOTICE

International application No.

PCT/EP00/07910

International filing date (day/month/year)

11 August 2000 (11.08.00)

Priority date (day/month/year)

17 August 1999 (17.08.99)

Applicant

EXXONMOBIL RESEARCH AND ENGINEERING COMPANY et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:

US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

CA, EP, JP, SG

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 22 February 2001 (22.02.01) under No. WO 01/12762

REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a **demand for international preliminary examination** must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the **national phase**, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO
 34, chemin des Colombettes
 1211 Geneva 20, Switzerland

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Authorized officer

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Continuation of Form PCT/IB/308

NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF
THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

Date of mailing (day/month/year) 22 February 2001 (22.02.01)	IMPORTANT NOTICE
Applicant's or agent's file reference P1999S004	International application No. PCT/EP00/07910

The applicant is hereby notified that, at the time of establishment of this Notice, the time limit under Rule 46.1 for making amendments under Article 19 has not yet expired and the International Bureau had received neither such amendments nor a declaration that the applicant does not wish to make amendments.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



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Lawton [GB/US]; 18 Saddler Drive, Medford, NJ 08055
(US).

(21) International Application Number: **PCT/EP00/07910**

(74) Agents: **DEW, Melvyn, John et al.**; ExxonMobil Chemical Europe Inc., P.O. Box 105, B-1830 Machelen (BE).

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Published:

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- *Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.*

(71) Applicant (*for all designated States except US*): **EXXONMOBIL RESEARCH AND ENGINEERING COMPANY** [US/US]; 1545 Route 22 East, Clinton Township, Annandale, NJ 08801 (US).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(72) Inventor; and

(75) Inventor/Applicant (*for US only*): **HOLT, David, Gary,**



WO 01/12762 A1

(54) Title: CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS

(57) Abstract: Lubricating oil formulations comprising base oil, such sulfur-phosphorous anti-wear/extreme pressure agents and such hindered phenol antioxidants which anti-wear/extreme-pressure agents and hindered phenolic antioxidants are prone to react and form crystals wherein the base oil is characterized as having a saturates content of less than 99 % which base oil is stabilized against the above mentioned crystal formation by the addition of a minor amount of a high molecular weight di- or polycarboxylic acid anhydride, or mixture thereof.

CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS

5

This invention relates to lubricating oil based on base stocks having
10 less than 99 wt% saturates content and containing one or more sulfur-phosphorus containing anti-wear/extreme pressure additives and one or more hindered phenol anti oxidants which combination are prone to crystal formation, wherein the formation of crystals is reduced or eliminated by the use of a crystallization suppressant.

15

Lubricating oils containing various antioxidants or esters or fatty acid amides or sulfur-phosphorus additives in combination with phenols are known in the literature.

20

U.S. Patent 5,167,844 is directed to a formulation comprising a base oil, at least one sulfur phosphorus containing compound, at least one amine and at least one hindered phenol.

25

JP 07034078 is directed to a hydraulic oil comprising mineral oil with an aromatic content of up to 1.5 wt% and a phenolic and aminic anti-oxidant, an alkenyl succinic acid imide rust inhibitor and a phosphoric acid type anti wear agent.

30

U.S. Patent 5,580,483 is directed for lubricating a refrigeration system compressor using a break-in lubricating oil which is an ester type oil. Additionally an adipate, phthalate, azelate, sebacate, trimellitate can also be present as well as tri hydrocarbyl phosphate, corrosion inhibitors such as alkali and/or alkaline earth metal sulfonate, antioxidants such as aminic or phenolic antioxidants and metal deactivators such as triazoles.

5

WO 97/14776 is directed to hydraulic oils comprising base oils combined with an amine antioxidant, a phenolic antioxidant, a phosphate ester and a fatty acid amide and/or polyhydric alcohol ester.

10 U.S. Patent 5,773,393 is directed to a composition comprising at least 70 wt% oil of lubricating viscosity and an amount effective to inhibit metal corrosion of a soluble additive comprising (a) at least one amide compound of a mono- or polycarboxylic acid or reactive derivative thereof and (b) at least 0.5 equivalents of at least one primary or secondary amine per mole of amide provided that when (a) is an amide of a dicarboxylic acid and the amine is an alkanol amine the mixture contains more than 0.5 equivalent of the amine (b) per equivalent of the amide.

20 The present invention is directed to a lubricating oil formulation having a reduced potential for the formation of crystals comprising a major amount of a lubricating oil base stock having less than about 99 wt% saturates content, preferably less than about 98 wt% saturates content, and a minor amount of additives comprising a mixture of sulfur-phosphorus containing anti-wear/extreme pressure additive, hindered phenol antioxidant and one or more high molecular weight di-, or polycarboxylic acid, anhydride or mixture thereof such as polyolefin succinic acid/anhydride, and to a method for reducing crystal formation in lubricating oil formulations comprising base oil having less than about 99 wt% saturates content, preferably less than about 98 wt% saturates content, and containing sulfur phosphorus anti-wear/extreme pressure additive and hindered phenolic anti-oxidant wherein the crystals are attributed to the interaction between the sulfur phosphorus containing anti-wear/extreme pressure agent and the hindered phenol by adding to said lubricating oil a minor effective amount of one or more high molecular weight di- or polycarboxylic acid or

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- 5 anhydride such as polyolefin succinic acid/poly olefin succinic anhydride and/or mixtures thereof.

10 The lubricating base oil is any oil of lubricating oil viscosity having less than about 99 wt% saturates content, preferably less than about 98 wt%
10 saturates content.

15 Lubricating oils meeting this criterion are any natural mineral or petroleum based lubricating oils derived from crude oil, tar sands, shale oil, etc., such that they contain a quantity of unsaturation resulting in a saturates content of less than of 99%, or a mixture of natural mineral or petroleum based lubricating oils in combination with a base oil or oils having a saturates content of greater than 99 wt%, e.g. hydrocarbon oils such as white oils and/or severely hydrotreated, hydrocracked mineral oils, or synthetic oils such as poly alpha olefins, esters, isomerized wax or isomerized Fischer-Tropsch wax, the
20 combination or mixture of such oils being characterized as having less than about 99 wt% saturates. Saturates content, for the purposes of this specification, is a measure of the absence of aromatic species, and was determined by high pressure liquid chromatography (HPLC) according to method IP 368, except where otherwise expressly indicated.

25

The lubricating oil base stocks useful in the present invention have the typical lubricating oil viscosity, usually possessing kinematic viscosities in the range of about 1.5 to 500 mm²/s at 100°C, preferably 5 to 120 mm²/s at 100°C.

30

Mineral or petroleum based lubricating oil base stocks can be derived from paraffinic, naphthenic and mixed base crudes. Conventional refinery techniques include distillation, solvent and/or catalytic dewaxing,

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- 5 solvent extraction, hydrofinishing, hydrocracking, vis breaking, deasphalting,
etc.

Synthetic lubricating oils that can be used include esters of di- and tri-basic acids, reacted with linear or branched aliphatic alcohols such as C₆-C₁₅ alcohols, such as di-2-ethylhexyl sebacate, phthalic ester esters of glycols such as C₁₃ oxo acid diester or tetraethylene glycol, or complex esters such as one formed from 1 mole of sebacic acid and 2 moles of tetraethylene glycol and 2 moles of 2-ethylhexanoic acid. Other synthetic oils that can be used include synthetic hydrocarbons such as alkyl benzenes, e.g., alkylate bottoms from the alkylation of benzene with tetrapropylene, or the copolymers of ethylene and propylene; silicone oils, e.g., ethyl phenyl polysiloxanes, methyl polysiloxanes, etc.; polyglycol oils, e.g., those obtained by condensing butyl alcohol with propylene oxide; carbonate esters, e.g., the product of reacting C₆ oxo alcohol with ethyl carbonate to form a half ester followed by reaction of the latter with tetraethylene glycol, etc.. Other suitable synthetic oils include the polyphenyl ethers, e.g., those having from about 3 to 7 ether linkages and about 4 to 8 phenyl groups.

Other suitable oils are the polyol ester oils made by reacting an aliphatic polyol with carboxylic acid. Aliphatic polyols contain from 4 to 15 carbon atoms and has from 2 to 8 esterifiable hydroxyl groups. Examples of polyols are trimethylolpropane, pentaerythritol, dipentaerythritol, neopentyl glycol, tripentaerythritol and mixtures thereof. The carboxylic acid reactant is selected from aliphatic monocarboxylic acid or mixtures of aliphatic mono carboxylic acids or mixtures of aliphatic mono- and di-carboxylic acids. The carboxylic acids contain 4 to 12 carbons and include straight and branched chain carboxylic acids.

- 5 -

5 Included in the group of synthetic oils are those recovered from tar
sands, shale oil, light hydrocarbons produced via, for example, the Fischer-
Tropsch process for converting synthesis gas (CO and hydrogen) into hydro-
carbons, wax isomerate oils produced by the catalytic hydroisomerization of
natural petroleum waxes (i.e., slack wax) or synthetic waxes (i.e., Fischer-
10 Tropsch waxes) or mixtures of such waxes. See USP 5,059,299 and USP
5,158,671 for description of wax isomerization and the oils produced thereby.
Other synthetic oils include the polyolefins such as polybutene, polyisobutenes
and especially the polyalphaolefins, i.e., fluids formed by the oligomerization of
at least one 1-alkane hydrocarbon having from 6 to 20 carbons, preferable 8 to
15 16 carbons, more preferably 8 to 12 carbons.

20 Regardless of the source of the oil, for the purposes of the present
invention, the lube oil base stock, be it a single oil or a mixture of oils, is
characterized as having a saturates content of less than about 99%, preferably
less than 98 wt%.

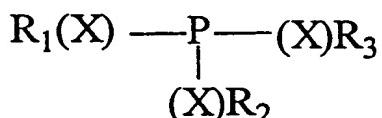
Sulfur-phosphorus containing anti-wear/extreme pressure additives
are well known in the industry, and are materials containing both sulfur and
phosphorus in the same molecule. For the purpose of the present specification,
25 and appended claims sulfur-phosphorus containing anti wear, extreme pressure
additives are those which react with hindered phenols to produce crystals. Those
skilled in the formulation art can readily determine without expenditure of
inventive effort, whether a particular sulfur-phosphorus containing anti-
wear/extreme pressure agent reacts with hindered phenol anti-oxidant to produce
30 crystals. If it does not, it is not within the scope of this invention. Any sulfur-
phosphorus containing anti-wear/extreme pressure agent which is found to react
with hindered phenol antioxidant to produce crystals in the subject base oil is
within this invention and formalities containing such agents and phenolic

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5 antioxidants will be beneficially affected is evidenced by reduction on
elimination of crystal formation by the addition of the high molecular weigh di-
or poly carboxylic acid, anhydride or mixture thereof, as shown below, provided
such carboxylic acid, anhydride or mixture thereof is used in an amount of at
least about 0.0013 wt% for each 1 ppm phosphorus attributable to the sulfur-
10 phosphorus containing anti-wear/extreme pressure agent.

Sulfur-phosphorus anti-wear/extreme pressure additives which
interact with hindered phenols to produce crystals are exemplified by, but not
limited to, materials of the type:

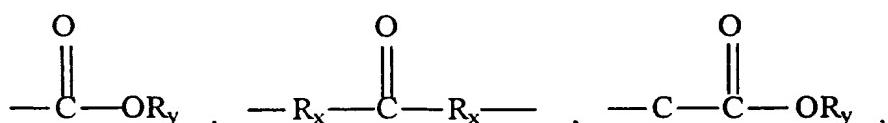
15



wherein R₁, R₂ and R₃ are independently hydrogen or hydrocarbyl provided at
least one is hydrocarbyl so as to render the material oil soluble and X is sulfur.

20

The hydrocarbyl groups preferably contain form 1 to 40 carbons and
are aromatic and/or aliphatic groups and include aryl alkyl and alkaryl and
aralkyl and heteroatom substituted aromatic and aliphatic group, the heteroatom
substitutents being sulfur, nitrogen or oxygen substitutented as such into the
25 hydrocarbon skeleton or as sulfur, oxygen or nitrogen containing moiety, e.g.,
—OR_y, —SH, —SO₂H, —N(R_y)₂, —C—R_xOR_y,

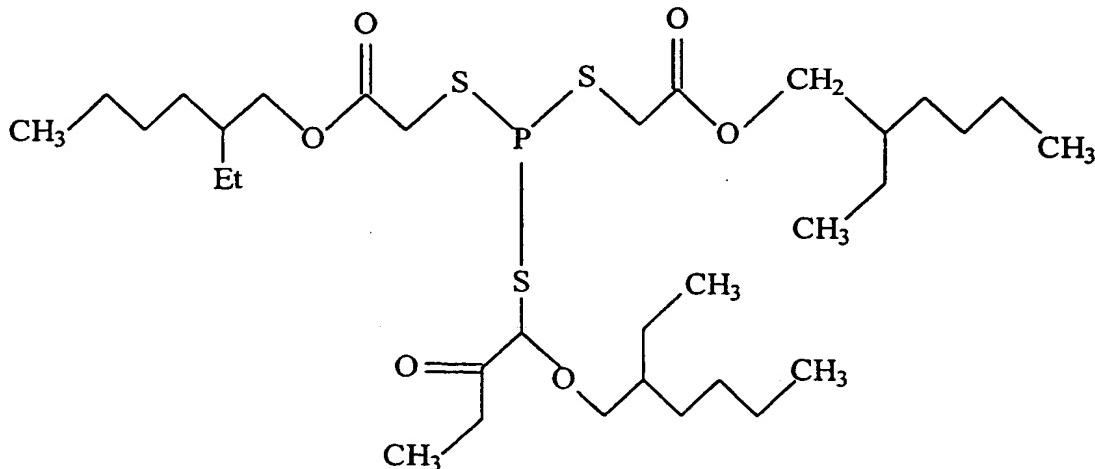


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5 etc., and mixtures thereof substituted onto or into the hydrocarbon backbone,
wherein R_X is C₁-C₂₀ hydrocarbyl or hydrocarbylene group and R_y is hydrogen
or a C₁-C₂₀ hydrocarbyl or hydrocarbylene.

Such sulfur-organo phosphorus containing anti-wear/extreme
10 pressure agent is typically used at a concentration sufficient to provide of from
about 2 ppm to 320 ppm phosphorus, preferably 40 ppm to 200 ppm phosphorus,
most preferably about 80 ppm to 130 ppm phosphorus.

An example of a sulfur phosphorus anti-wear/extreme pressure
15 additive which has been found to react with hindered phenols to form crystals is
a material is 2-ethylhexyl 10-ethyl-4-[[2-[(2 ethylhexyl)-oxyl]-2-oxoethyl] thio]-
7-oxo-8-oxa-3,5-dithia-4-phospha tetradecanoate, CAS # 83547-95-9. Based on
the name and the CAS number, it is believed this material has the following
structure:



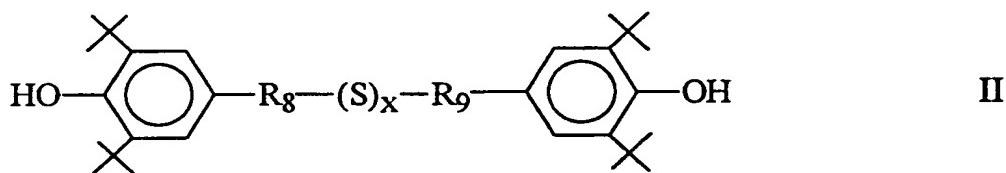
20

It must be noted that for the purposes of the present invention metal
dihydrocarbyldithiophosphate (metal DDP) or ashless DDP do not fall within the
above definition of sulfur-phosphorus containing anti-wear/extreme pressure

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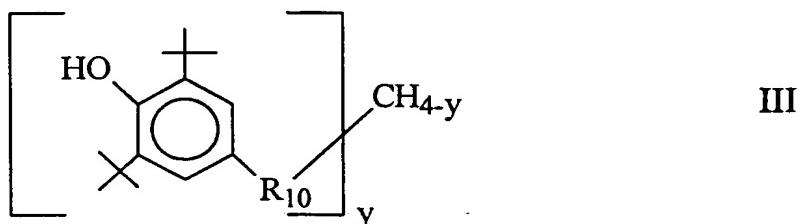
- 5 additive because it has been found that they do not form crystals when combined
with hindered phenols in base oils.

Hindered phenolic anti oxidants are also well known in the industry.
Such materials include by way of example and not limitation 2,6-di-t-butyl
10 phenol, 2,6-di-t-butyl alkylated phenol where the alkyl substituent is hydrocarbyl
and contains between 1 and 20 carbon atoms, such as 2,6-di-t-butyl-4-methyl
phenol, 2,6-di-t-butyl-4-ethyl phenol, etc., or 2,6-di-t-butyl-4-alkoxy phenol
where the alkoxy substituent contains between 1 and 20 carbons such as 2,6-di-t-
butyl-4-methoxyphenol; materials of the formula



15

where X is zero to 5, R₈ and R₉ are the same or different and are C₁-C₂₀
hydrocarbyl which may contain oxygen or sulfur or be substituted with oxygen
or sulfur containing groups; and materials of the formula

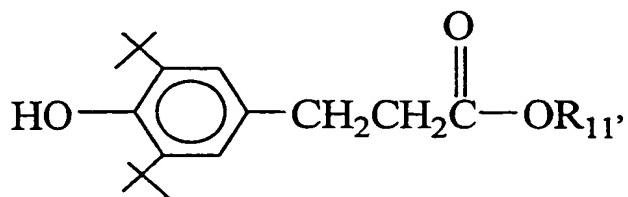


20

where y is 1 to 4 and R₁₀ is a C₁ to C₂₀ hydrocarbyl which may contain oxygen
sulfur or nitrogen or be substituted with oxygen, sulfur or nitrogen containing
groups such as 2,6 di tert butyl α dimethylamino P-cresol,

25

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5 IV

wherein it is believed R₁₁ is C₈C₁₇ (CAS # 125643-61-0), and mixtures of such phenolic type antioxidants.

10 Preferably the phenolic anti-oxidant contains an ester group, such as in formula IV above.

15 Phenolic type anti oxidants are typically used at a concentration of from about 0.01 to 2.0 wt%, preferably about 0.1 to 1.0 wt%, most preferably about 0.3 to 0.5 wt%, based on active ingredient.

20 In order to prevent or at least minimize the formation of crystals in lubricating oils based on base stock having less than 99% saturates preferably less than 98 wt% saturates and containing a mixture of sulfur-organo phosphorus anti-wear/extreme pressure additive and phenolic anti-oxidant, wherein the 25 sulfur phosphorus containing anti-wear/extreme pressure agent interests with the hindered phenol to produce crystals a minor, crystal preventing effective amount of a high molecular weight carboxylic acid, anhydride or mixture thereof is added to the lubricating oil formulation.

25 The carboxylic acid or anhydride can be any high molecular weight acid such as di- or polycarboxylic acid, anhydride or mixture thereof of molecular weight of about 300-5000. Such acids, anhydrides or mixtures thereof include polyhydrocarbylene substituted di- or polycarboxylic acids or anhydrides 30 wherein the poly hydrocarbylene group has a molecular weight in the range 300 to 5000, preferably 750 to 2000, most preferably 900 to 1000 (e.g.,

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5 polyisobutylene) and wherein the carboxylic group is, e.g., succinic or maleic acid, anhydride or mixture thereof.

Poly hydrocarbylenes are homopolymer or interpolymers of polymerizable olefin group containing monomers having from 2 to 16 carbons.

10 Interpolymers are those made using two or more different olefinic groups containing monomer including monomer such as styrenes. Poly hydrocarbylene homo and interpolymers are well known in the literature and to those skilled in the art and need not be further described herein.

15 Preferably the carboxylic acid or anhydride or mixture thereof used is polyalkylene succinic or maleic acid, anhydride, or mixtures thereof, most preferably polyisobutylene (PIB) succinic acid, anhydride or mixtures thereof wherein the PIB group has a molecular weight of about 900 to 1000.

20 Such high molecular weight carboxylic acids, anhydrides are employed in an amount in the range of about 0.0026 to 0.8 wt%, preferably about .08 to 0.4 wt%, most preferably about 0.12 to 0.24 wt%, based on active ingredients.

25 In general, at least 0.0013 wt% of high molecule weight carboxylic acid, anhydride or mixture thereof is used for each 1 ppm phosphorous from the sulfur-organo phosphorus anti-wear/extreme pressure agent.

EXAMPLES

30 Example 1

This example (Table 1) is presented to show that, in a base stock having a saturates content of less than 99 wt%, the combination of a sulfur-

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- 5 phosphorous anti-wear/extreme pressure agent with a hindered phenol results in crystal formation while the combination of a sulfur free phosphate extreme pressure agent and hindered phenol does not result in crystal formation.

TABLE 1

				<u>Crystals at 3 months</u>
Base oil (1)	+	.55 wt% sulfur-phosphorus extreme pressure agent (2)	+ 0.4 wt% hindered phenol (3)	yes
Base oil (1)	+	.4 wt% hindered phenol	+ .55 wt% sulfur free phosphate EP agent (4)	no

10

- (1) solvent refined base oil, about 88% saturates 150 SN oil
- (2) sulfur phosphorus extreme pressure agent CAS #83547-95-9 which is 60% sulfur-phosphorus component active ingredient (also contained C₄-C₈ diphenyl amine as balance of additive)
- (3) 100% active ingredient, CAS # 125643-61-0
- (4) 100% active ingredient, isopropylated triaryl phosphate

15

The resulting lubricant had a phosphorus content of 120ppm by weight, measured according to standard test ASTM D5185-97, attributable to the sulphur-phosphorus extreme pressure agent (which was the sole phosphorus-containing component contained in the lubricant formulation)

20

Example 2

This example (Table 2) is presented to show that crystal formation is eliminated in formulations normally exhibiting crystal formation by the addition of high molecular weight anhydride but that crystal formation is not eliminated by the addition of high molecular weight anhydride-poly amine dispersant, or by the addition of esters. All formulations tested in this example further contained typical pour point depressants, anti-rust agent and an amino para cresol antioxidant.

30

TABLE 2

Base oil (1)	+ .55 wt% sulfur-phosphorus EP agent (2)	Crystals at 3 months	
		0.4 wt% hindered phenol (3)	+
(“)	(“)	(“)	+
(“)	(“)	(“)	PIBSA + PAM (4)
(“)	(“)	(“)	+ Esters (5)
(“)	(“)	(“)	+ PIBSA (6)
(“)	(“)	(“)	+ PIBSA (7)
			no

- 1) Base oil, a 50/50 mixture of 150 N (88% saturates) and 400 N (about 78% saturates).
- 2) See Table 1.
- 3) See Table 1.
- 4) PIBSA-PAM was tested at concentration of from 0.05 to .4 wt% and at all concentrations used crystals formed within the three month time period of the test.
- 5) Esters tested were di iso nonyl phthalate at 0.05 to 4 wt%; di iso-tridecyl adipate at .1 to .5 wt%; C₆ and C₁₃ phthalate at .5 wt%. None were effective at eliminating crystal formation during the three month time period of the test.
- 6) PIBSA is polyisobutylene succinic anhydride, having a polyisobutylene molecular weight of 950. When used at .04 wt% and .08 wt% active ingredient level, it did not eliminate crystal formation.
- 7) PIBSA (of note 6) at .16 wt% and .32 wt% active ingredient level eliminated crystal formation.

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5

Example 3

This example (Table 3) is presented to show the effect of base stock saturation on the suppression of crystal formation when using PIBSA in combination with sulfur phosphorus extreme pressure agent and hindered phenol.

TABLE 3

5

Crystals at

Base oil (1)	+ 0.55 wt% sulfur phosphorus extreme pressure agent (2)	+ phenol (3)	+ 0.4 wt% hindered phenol (3)	+ 0.16 wt% PIBSA (4)	- 14 -
Base oil (5)	(“)	(“)	(“)	(“)	no
Base oil (6)	(“)	(“)	(“)	(“)	no
Base oil (7)	(“)	(“)	(“)	(“)	no
Base oil (8)	(“)	(“)	(“)	(“)	cloudy
Base oil (9)	(“)	(“)	(“)	(“)	cloudy
Base oil (10)	(“)	(“)	(“)	(“)	cloudy
Base oil (10)	(“)	(“)	(“)	(“)	yes

(1) See Table 1.

(2) See Table I.

(3) See Table 1.

10 (4) PIBSA is polyisobutylene succinic anhydride. Polyisobutylene molecular weight 950.

(5) 150 N. about 80% saturates.

(6) 150N FDA C grade white oil about 80% saturate (by clay-gel analysis - ASTM D 2007).

(7) Hydrocracked 90 N. about 92% saturates.

(8) 150 N FEDA A grade white oil. 100% saturates

(g) Hydrocracked 150 N. about 99.9% saturates.

(10) PAO-6, 100% saturates.

10

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5

From this it is seen that in base oils having less than 99% saturates and containing mixtures of sulfur-phosphorus extreme pressure agent and hindered phenol, which are prone to crystal formation, crystal formation is suppressed on adding PIBSA where as in base oils of essentially 100% saturates
10 content even addition of PIBSA failed to prevent crystal formation and even increasing PIBSA concentrate to 0.8 wt% (active ingredient) did not prevent crystal formation.

5 CLAIMS :

1. A lubricating oil of reduced crystal formation potential attributable to the interaction of sulfur phosphorus containing anti-wear/extreme pressure agents and hindered phenolic antioxidants comprising a major amount of a base oil of lubricating viscosity and having less than about 99 wt% saturates content, and a minor amount of additive comprising a sulfur-phosphorus containing anti-wear/extreme pressure additive, a hindered phenol antioxidant and a high molecular weight di- or poly- carboxylic acid, anhydride or mixture thereof provided at least 0.0013 wt% high molecular weight carboxylic acid, anhydride or mixture thereof is present for each 1 ppm phosphorus attributable to the sulfur phosphorus containing anti-wear/extreme pressure agent.
2. The lubricating oil of claim 1 wherein the sulfur-phosphorus anti-wear/extreme pressure agent is in an amount sufficient to provide about 2 ppm to 320 ppm phosphorus, the hindered phenol antioxidant is at a concentration of from about 0.01 to 2.0 wt% based on active ingredient and the high molecular weigh di- or poly-carboxylic acid is at a concentration of in the range of about 0.0026 to 0.8 wt% based on active ingredient.
3. The lubricating oil of claim 1 or 2 wherein the sulfur-phosphorus containing anti-wear/extreme pressure agent is in an amount sufficient to provide from 40 ppm to 200 ppm phosphorus.
4. The lubricating oil of claim 1 or 2 wherein the sulfur-phosphorus containing anti-wear/extreme pressure agent is in an amount sufficient to provide from 80 ppm to 130 ppm phosphorus.

5 5. The lubricating oil of any preceding claim wherein the hindered phenol is at a concentration of about 0.1 to 1.0 wt% based on active ingredient.

o
6. The lubricating oil of claim 2, 3 or 4 wherein the hindered phenol is at a concentration of about 0.3 to 0.5 wt% based on active ingredient.

10 7. The lubricating oil of claim 2, 3 or 4 wherein the high molecular weight di- or poly-carboxylic acid, anhydride or mixture thereof is at a concentration of about 0.08 to 0.4 wt% based on active ingredient.

15 8. The lubricating oil of any preceding claim wherein the high molecular weight di- or poly-carboxylic acid anhydride or mixture thereof is at a concentration of about 0.12 to 0.24 wt% based on active ingredient.

20 9. The lubricating oil of any preceding claim wherein the high molecular weight di- or poly-carboxylic acid, anhydride mixture thereof is a polyhydrocarbylene substituted di- or poly-carboxylic acid, anhydride or mixture thereof wherein the polyhydrocarbylene group has a molecular weight in the range 300 to 5,000.

25 10. A method for reducing crystal formation in lubricating oil containing a mixture of sulfur phosphorus anti-wear/extreme pressure agent and hindered phenols antioxidant wherein the sulfur-phosphorus anti-wear/extreme pressure agent interacts with the phenolic antioxidant to produce crystals, such method comprising adding to a major amount of a base oil of lubricating viscosity having a saturates content of less than 99 wt%, a minor amount of additives comprising a sulfur-phosphorus containing anti-wear/extreme pressure agent a hindered phenol antioxidant and a high molecular weigh di- or poly-carboxylic acid, anhydride or mixture thereof provided at least 0.0013 wt% of

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- 5 the high molecular weight di- or poly-carboxylic acid, anhydride or mixture thereof is used for each 1 ppm phosphorus attributable to the sulfur-phosphorus containing anti-wear/extreme pressure agent.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/07910

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10M163/00 // (C10M163/00, 159:12, 129:42, 129:93)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 458 495 A (PANZER JEROME ET AL) 29 July 1969 (1969-07-29) column 1, line 33 -column 1, line 41 column 2, line 53 -column 3, line 29 example 1 column 6, line 63 -column 8, line 29 -----	1-10
A	US 2 766 207 A (JOHN PATRICK MCDERMOTT ET AL) 9 October 1956 (1956-10-09) the whole document -----	1-10



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

& document member of the same patent family

Date of the actual completion of the international search

30 January 2001

Date of mailing of the international search report

05/02/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl.
Fax: (+31-70) 340-3016

Authorized officer

Perakis, N

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/07910

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3458495	A 29-07-1969	NONE	
US 2766207	A 09-10-1956	NONE	

PATENT COOPERATION TREATY
PCT
INTERNATIONAL PRELIMINARY EXAMINATION REPORT
(PCT Article 36 and Rule 70)

Applicant's or agent's file reference P1999S004	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/EP00/07910	International filing date (day/month/year) 11/08/2000	Priority date (day/month/year) 17/08/1999
International Patent Classification (IPC) or national classification and IPC C10M163/00		
Applicant EXXONMOBIL RESEARCH AND ENGINEERING COMPANY et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 4 sheets, including this cover sheet.

This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

Date of submission of the demand 13/03/2001	Date of completion of this report 05.04.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Perakis, N Telephone No. +49 89 2399 8355



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/EP00/07910

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):
Description, pages:

1-15 as originally filed

Claims, No.:

1-10 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- the language of publication of the international application (under Rule 48.3(b)).
- the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- contained in the international application in written form.
- filed together with the international application in computer readable form.
- furnished subsequently to this Authority in written form.
- furnished subsequently to this Authority in computer readable form.
- The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- the description, pages:
- the claims, Nos.:
- the drawings, sheets:

5. This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/EP00/07910

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-10
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-10
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-10
	No:	Claims	

2. Citations and explanations
see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/EP00/07910

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

The present international application concerns a lubricating composition comprising a base oil and various additives. The base oil is one containing less than 99 wt % of saturates. The additives are a specific mixture of a S/P antiwear additive and a hindered phenol antioxidant additive which are so selected that they react. The composition also contains an additional additive which is a di/poly acid anhydride. This additive stabilizes the base oil in view of the crystal formed by the reaction product of the two previous additives.

The subject-matter claim is novel over the state of the art. D1: US-A-3458495 (cf. passages cited in the International Search Report) discloses the reaction between a P/S antiwear additive and a hindered phenol. It does not however disclose the specific combination of the previous additives with a di/poly acid anhydride.

The technical problem this combination solves is the stabilisation of the base oils with a saturates content of less than 99 wt%. The solution of the set technical problem does not seem to be obvious since it does not derive from the state of the art and does not seem to belong to the general technical knowledge of the person skilled in the art.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference P1999S004	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/EP 00/07910	International filing date (day/month/year) 11/08/2000	(Earliest) Priority Date (day/month/year) 17/08/1999
Applicant EXXON RESEARCH AND ENGINEERING COMPANY et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
 - the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
- b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :
 - contained in the international application in written form.
 - filed together with the international application in computer readable form.
 - furnished subsequently to this Authority in written form.
 - furnished subsequently to this Authority in computer readable form.
 - the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
 - the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. Certain claims were found unsearchable (See Box I).

3. Unity of invention is lacking (see Box II).

4. With regard to the **title**,

- the text is approved as submitted by the applicant.
- the text has been established by this Authority to read as follows:

CRYSTAL FORMATION INHIBITION IN LUBRICATING COMPOSITIONS

5. With regard to the **abstract**,

- the text is approved as submitted by the applicant.
- the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No.

- as suggested by the applicant.
- because the applicant failed to suggest a figure.
- because this figure better characterizes the invention.

None of the figures.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/EP 00/07910

B x III TEXT OF THE ABSTRACT (Continuation of item 5 of the first sheet)

Lubricating oil formulations comprising a base-oil, such a sulfur-phosphorous anti-wear/extreme pressure agents and such hindered phenol antioxidants which anti-wear/extreme-pressure agents and hindered phenolic antioxidants are prone to react and form crystals wherein the base oil is characterized as having a saturates content of less than 99% which base oil is stabilized against the above mentioned crystal formation by the addition of a minor amount of a high molecular weight di- or polycarboxylic acid anhydride, or mixture thereof.

INTERNATIONAL SEARCH REPORT

International Application No

EP 00/07910

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10M163/00 // (C10M163/00, 159:12, 129:42, 129:93)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 458 495 A (PANZER JEROME ET AL) 29 July 1969 (1969-07-29) column 1, line 33 -column 1, line 41 column 2, line 53 -column 3, line 29 example 1 column 6, line 63 -column 8, line 29 -----	1-10
A	US 2 766 207 A (JOHN PATRICK MCDERMOTT ET AL) 9 October 1956 (1956-10-09) the whole document -----	1-10



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

° Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

30 January 2001

Date of mailing of the international search report

05/02/2001

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Authorized officer

Perakis, N

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

EP 00/07910

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3458495	A 29-07-1969	NONE	
US 2766207	A 09-10-1956	NONE	